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Applicant (Assignee of Actual Inventor)

SOCIÉTÉ GÉNÉRALE MÉTALLURGIQUE DE HOBOKEN.

Actual Inventor

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Application and Complete Specification

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No drawing.

### COMPLETE SPECIFICATION.

#### "Improvements in or relating to the treatment of materials containing tantalum and/or niobium."

We, SOCIÉTÉ GÉNÉRALE MÉTALLURGIQUE DE HOBOKEN, of Hoboken-lez-Anvers, Belgium, a Belgian Limited Company, metal refiners, hereby declare this invention and the manner in which it is to be performed to be fully described and ascertained in and by the following statement:—

This invention relates to an improved process for the recovery of tantalum and/or niobium from their ores, or from metallurgical by-products such as those in which the said elements may be found in the form of oxide compounds, free or combined.

This invention has for its object the recovery by an economical process of tantalum and/or niobium in the form of metals or of alloys.

One feature of the present invention consists in the combination of a reducing operation by means of one or several of the metals aluminium, calcium, silicon, magnesium, of the primary material, with a subsequent reducing operation in an electric furnace of the slag obtained from the first said reducing operation, so as to obtain on the one hand a tantalum and/or niobium alloy and on the other hand a slag poor in tantalum and niobium.

The first reducing operation in the presence of one or more of the metals, Al, Ca, Si, Mg may be carried out without other external heat than that required for starting the reaction or it may be carried out in an electric furnace with application of external heat. The said first reducing operation is carried out in the presence of the metals with which it is desired to alloy the tantalum and/or niobium, for instance with iron. If the said metal is not present in a sufficient amount in the primary material, it is added in the metal or oxide form, so as to obtain the required tantalum and/or niobium alloy, for instance ferro-tantalum and/or ferro-niobium.

The second reducing operation, of the slag comparatively rich in Ta and/or Nb in an electric furnace, may be carried out in the presence of reducing agents, such as Ca, Al, Si, Mg used either separately, or together, or by two.

According to another feature of the present invention, the primary material is subjected to a selective reducing operation in the presence of alloys of Nb and/or Ta or of ferro-tantalum and/or ferro-niobium with one or several of the following reducing metals: Ca, Al, Si, Mg.

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In this way it is possible to concentrate the Ta in a product poor in Nb; this product may serve as the primary material for the manufacture of commercial tantaliferous products.

This feature of the process may be carried out as follows:

1. A ferro-tantalum and ferro-niobium alloy containing one or several of the metals Ca, Si, Al, Mg is melted in an electric furnace, of the Héroult type for instance, in the presence of the primary material containing oxides of Ta and/or Nb, with the addition of a flux such as CaO, SiO<sub>2</sub>, fluor-spar, etc. A slag is thus formed which acts upon the reducing metals of the ferro-Ta/Nb. The niobium is reduced and passes into an alloy, whilst the oxides of the reducing metals pass into the slag. The slag is tapped as soon as it is exhausted or sufficiently poor in Nb.

A second slag containing the oxides of Ta + Nb is then produced from the ferro-Ta + Nb and tapped after being deprived substantially of its Nb. This operation is continued as long as there is a sufficient amount of reducing metals left in the ferro-Ta + Nb.

2. The raw material containing the oxides of Ta and/or Nb is mixed with a flux, and with a ferro-tantalum-niobium containing the reducing elements Ca, Mg, Al, Si. The reducing metals act during the melting operation upon the oxide of Nb of the primary material. The slag is tapped, poor in Nb, and the mixture is charged again.

3. The raw material containing the oxides of Ta and/or Nb is reduced by Ca, Al, Mg, Si taken separately or together.

4. The ferro-Ta-Nb obtained, containing a certain amount of reducing metals, is then refined after having tapped the last slag. For this purpose, the raw material is charged with or without addition of flux. The refining slag, poor in niobium is removed and the refining operation is continued as long as there is left a sufficient amount of reducing metals in the ferro-Ta-Nb.

5. According to another feature of the invention, the primary material is subjected to a reducing operation with one or more of the elements Al, Si, Ca, Mg, alloyed or combined with one or more metals as

characterized below, in order to substantially separate the whole or the major part of the niobium or tantalum when one alone of said elements is present, or the niobium and tantalum together, when both are present, in the form of alloy.

The metals which are added to the reducing agents are such metals whose alloying or combination reaction with the aforesaid reducing agents develops heat so that the metallo-thermic power of a given quantity of these reducing elements is smaller in the obtained alloy or combination than the same quantity in the elementary state. Among these metals, nickel, cobalt, iron, are more particularly contemplated.

The raw material containing the tantalum and/or niobium may be subjected inside any suitable metallurgical apparatus, for instance in an electric furnace, to a reducing operation, with one or more of the elements Al, Si, Ca, Mg, alloyed or combined to nickel and/or cobalt and/or iron.

In this way it is possible to obtain:

1. An alloy containing the major part of the reduced tantalum and/or niobium alloyed to the metal which has been used for reducing the thermal activity of the reducing agent, that is in the particular case alloyed to one or several of the elements nickel, cobalt, iron;

2. A slag rich in tantalum and/or niobium, which may be treated by any known process for the recovery of tantalum and/or niobium.

According to another feature of the invention, the primary material is subjected to a selective reduction in the presence of one or several of the following reducing elements: Ca, Al, Si, Mg, alloyed or combined with one or more metals as characterized below, so as to obtain a product (alloy for instance) in which the ratio Nb/Ta is higher than that of the primary material.

The metals which are added to the reducing agents are such metals whose alloying or combination reaction with the aforesaid reducing agents develops heat so that the metallo-thermic power of a given quantity of these reducing elements is smaller in the obtained alloy or combination than the same quantity in the elementary state. Among these metals, nickel, cobalt, iron, are more particularly contemplated.

For carrying this feature of the invention into effect, the primary material containing tantalum and niobium may be subjected inside a suitable metallurgical apparatus, such as an electric furnace, to a selective reduction in the presence of one or more of the elements Si, Ca, Al, Mg, the thermic capacity of which has been diminished by alloying or combining them to other metals such as Ni, Co, Fe. The amount of reducing agent may be so calculated that the major part of the Nb contained by the primary material is reduced and passes into an alloy in which the ratio Nb/Ta is higher than that of the primary material. The other component elements of the alloy are the metal or metals used for lowering the thermic capacity of the reducing agent.

By the process according to the present invention, an alloy is obtained in which the ratio Nb/Ta is higher than that of the primary material, and also a slag is obtained in which the ratio Ta/Nb is higher than that of the primary material. The slag may be treated by any suitable known process with a view to recovering the tantalum and niobium.

Having now fully described and ascertained our said invention and the manner in which it is to be performed, we declare that what we claim is:—

1. A process for the treatment of materials containing tantalum and/or niobium, characterized in that the primary material is subjected to a reducing operation by means of one or several of the metals aluminium, calcium, magnesium, silicon, whilst the slag obtained from the said reducing operation is subjected to a reducing operation in an electric furnace so as to obtain on the one hand a tantalum and/or niobium alloy, and on the other hand a slag poor in tantalum and niobium.

2. A process for the treatment of materials containing tantalum and/or niobium, characterized in that the primary material

is subjected to a selective reducing operation in the presence of alloys of Nb and/or Ta or of ferro-tantalum and/or ferro-niobium with one or several of the following reducing metals: Ca, Al, Si, Mg.

3. A process for the treatment of materials containing tantalum and/or niobium, characterized in that the primary material is subjected to a reducing operation with one or more of the elements Al, Si, Ca, Mg, alloyed or combined with one or more metals whose alloying or combination reaction with the aforesaid reducing agents develops heat so that the metallo-thermic power of a given quantity of these reducing elements is smaller in the obtained alloy or combination than the same quantity in the elementary state.

4. A process for the treatment of materials containing tantalum and/or niobium, characterized in that the primary material is subjected to a selective reduction in the presence of one or several of the following reducing elements: Ca, Al, Si, Mg, alloyed or combined with one or more metals, whose alloying or combination reaction with the aforesaid reducing agents develops heat so that the metallo-thermic power of a given quantity of these reducing elements is smaller in the obtained alloy or combination than the same quantity in the elementary state.

5. A process as claimed in Claim 3 or 4, characterized in that nickel and/or cobalt and/or iron are used for alloying or combining to the reducing agents.

6. The improved process for the treatment of materials containing tantalum and/or niobium, substantially as described.

Dated this third day of August, 1937.

SOCIÉTÉ GÉNÉRALE MÉTALLURGIQUE DE HOBOKEN,

By their Patent Attorney,  
P. M. BONNERUP.

Witness—J. Dennis.